

(19) Japan Patent Agency (JP)

(10) **Public Patent bulletin**

(11) Public Patent Application Number

**Sho 63 - 91231**

(43) Date of being Public Showa 63 (1988), 21<sup>st</sup> April

(51) Int. Cl<sup>4</sup>.      Discrimination      Internal Arrangement

Number

Number

B 29 D 11/00

6660 - 4F

B 29 B 13/10

7206 - 4F

B 29 C 47/68

6660 - 4F

B 41 M 5/26

V - 7265 - 2H

// B 29 K 25:00

71:00

Investigations of Claims **Nil**

Number of Inventions **2**

(Total 9 Pages (Japanese))

Sho 63 - 91231

Page

---

(54) Title of the invention      Optical Element and  
Method      for  
Manufacture of the  
Same

(21) Patent Application Number      Sho 61 - 236769

(22) Date of Patent Application      Sho 61 (1986), 3<sup>rd</sup>  
October

(72) Inventor      Moriya Masahiko  
  
Aichi ken, Shin Ihama  
  
shi, Sokai machi, 5 ban,  
  
1 go.  
  
Sumitomo Kagaku Kogyo  
  
Kabushiki Kaisha Nai  
  
(Internal to Sumitomo  
  
Chemical Industries

Ltd.)

(72) Inventor

Takano Masatani

Aichi ken, Shin Ihama  
shi, Sokai machi, 5 ban,  
1 go.

Sumitomo Kagaku Kogyo  
Kabushiki Kaisha Nai  
(Internal to Sumitomo  
Chemical Industries  
Ltd.)

(72) Inventor

Maami Shinni

Aichi ken, Shin Ihama  
shi, Sokai machi, 5 ban,  
1 go.

Sumitomo Kagaku Kogyo  
Kabushiki Kaisha Nai  
(Internal to Sumitomo  
Chemical Industries

Ltd.)

(71) Patent Applicant

Sumitomo Kagaku Kogyo

Kabushiki Kaisha

(Sumitomo Chemical

Industries Ltd.)

Oosaka fu, Oosaka shi,

Higashi ku, Kitahama,

5 cho me, 15 ban chi.

(74) Representative Patent Attorney Moroichi Komori

(Besides one more name)

## A detailed description

### 1. Title of the invention

Optical Element and Method for Manufacture of the Same.

## 2. Scope of patent claims

- (1) The optical element made of resin that has foreign fine particles of more than 1  $\mu\text{m}$ , of less than 10,000 units/g.
- (2) The method for manufacture of optical element that has the following characteristics. Namely, in the method to manufacture an optical element by subjecting a resin composite formed from polymer component containing aromatic vinyl unimer units, as its main constituent and polyphenylene ether component, to fusion molding, foreign particles of more than 1  $\mu\text{m}$  present in the resin composite mentioned above are less than 10,000 units / g.
- (3) The manufacturing method described above in claim number (2) in which the resin composite mentioned above is the resin composite that is recovered after removing the foreign particles by filtering the solution formed by dissolving the resin

composite in an organic solvent.

- (4) The manufacturing method described above in claim number (3) in which the organic solvent is at least 1 type chosen from chloroform, benzene, toluene and xylene.
- (5) The manufacturing method described above in claim number (3) in which recovery of resin composite mentioned above is the deposition that is carried out by using poor solvent of the resin composite mentioned above from the solution mentioned above.
- (6) The manufacturing method described above in claim number (3) in which recovery of the resin composite is carried out by supplying the solution mentioned above to screw extrusion crusher and pelletization is carried out simultaneously while removing the solvent.
- (7) The manufacturing method described above in claim number (3) in which recovery of the resin composite

mentioned above is carried out by adding water to the solution mentioned above and azeotropically removing the organic solvent and thus, forming an aqueous slurry.

- (8) The manufacturing method described above in claim number (2) in which the resin composite mentioned above is the resin composite from which the foreign particles are removed by passing the solution state material through a sintered metal filter.
- (9) The method described above in claim number (8) in which device installed in end part of the screw extruder is used as sintered filter.

### **3. Detailed description of the invention**

#### **(Industrial applicability)**

The present invention relates to the optical element having less foreign particles.

For example, the present invention relates to the optical element of optical disk baseboards that carry out

recording and regeneration of information optically and optical cards, lenses, prisms etc.

**(Techniques of the past)**

Regeneration of image or music in which recorded information engraved by the fine roughness on the optical disk baseboard by using laser beam spot is detected and further, recording regeneration style in which regeneration of recorded information having high density is carried out by change in optical properties of the recording film set up on the surface of the baseboard, is attracting attention during the recent years.

Properties such as satisfactory dimension stability, optical uniformity and low birefringence (double refraction) are required besides transparency, for the material to be used as optical disk baseboard used in such recording and regeneration method.

Baseboard reproduced in large quantity at cheap cost can be molded by the use of resin material as optical disk



baseboard, however, it is well known that in most of the cases, molecular orientation takes place in the --- of resin and cooling process at the time of molding the disk baseboard and therefore, birefringence (double refraction) gets generated, which is a major disadvantage.

Actually, at the time of using a material as optical disk baseboard, birefringence (double refraction) measured by Celmon (?) Compensator method at 546 nm is required to be within the range from + 20 ~ - 20 nm.

The optical recording element formed from optically isotropic resins such that the birefringence (double refraction) becomes essentially zero, obtained by mixing 2 types of polymers that possess exactly opposite optical isotropy but are completely compatible, with the composition such that their optical anisotropy becomes exactly negative, has been presented in American Patent 4,373,065.

Furthermore, the system using polyphenylene ether and polystyrene as the polymers possessing exactly opposite optical anisotropy has been reported in public patents, and moreover, it has been reported that birefringence (double refraction) does not get generated in the film prepared from a mixture having a composition in which their optical anisotropy has been exactly negated, even by applying stress, in other words, in the case of applying stress to the polymer composite in solid state, birefringence (double refraction) does not get generated.

Furthermore, as regards these optical disk baseboards, material having less optical anisotropy by birefringence (double refraction), along with having sufficiently high carrier noise ratio (called as C / N ratio) at the time of regeneration of recording, is required.

An optical disk containing foreign substance having size of particle diameter of more than  $0.5\ \mu\text{m}$ , of less than  $1 \times 10^5$  units / g, in the optical disk baseboard made of

transparent resin obtained by injection molding, particularly acryl resin, has been presented in Patent number Sho 61 - 909345.

However, in the case of using a polymer containing methyl methacrylate as its main constituent known since past, in a baseboard, dimension stability is unsatisfactory due to high moisture absorbing property and therefore, warps, torsion get generated under high temperature environment, which is a disadvantage it possesses.

These disadvantages have been explained in detail in Nikkei Electronics (1982, 7<sup>th</sup> June, page 133) and therefore, aromatic polycarbonate having low moisture absorbance is being used as compact disk material used for sound (recording).

On the other hand, aromatic polycarbonate has high anisotropy and it mainly contains aromatic rings due to which lowering of birefringence (double refraction) of

the molded baseboard is difficult, and besides lowering of the molecular weight, molding conditions are being tested, however, birefringence (double refraction) ---- due to which baseboard having still low birefringence (double refraction) cannot be manufactured stably and moreover, manufacture of a baseboard having low birefringence (double refraction) and moreover, having larger diameter than that of compact disk used in sound (recording) is an extremely difficult condition.

Moreover, in order to reform dimension stability which is a drawback of polymer containing methyl methacrylate as its main constituent, copolymer of methyl methacrylate and aromatic vinyl unimer has been proposed in Patent number Sho 57 - 33446, Sho 57 - 162135 and Sho 58 - 88843.

However, copolymer formed with vinyl unimer containing aromatic rings, easily generates high birefringence (double refraction) and therefore, it cannot be practically used which is a current state of affairs.

**(Problems the invention solves)**

Optical element requires dimension stability and low birefringence (double refraction), however, among these also, in the case of optical disk baseboard, these requirements are stringent and moreover, sufficiently high C / N ratio at the time of recording and regeneration and less pit errors are required.

Furthermore, in the optical element of lenses, prisms also, resin material such as methacrylate resin is being used since past, however, material having still lower birefringence (double refraction) and excellent heat resistance, mechanical strength, dimension stability and less fine particles, is demanded for.

Furthermore, tests of making the disk baseboard of opto-magnetic type disks in case of which information can be deleted and re-written, lightweight by using plastic are being carried out.

In the case of opto-magnetic type disks, optical disk

in which ---- is difficult to occur with respect to light that is incident from the slanting direction, is required since polarized laser beam fixes black spots on the recording medium at the time of reading the recorded information and information is read by detecting slightly polarized light of laser beam by Ca (?) effect that comes back by reflection.

Moreover, at the time of writing, since heating is carried out by laser beam, material having high heat resistance is required to be used as optical disk baseboard.

With respect to these demands, optical disk baseboard formed from polymer component containing aromatic vinyl unimer as its main constituent and polyphenylene ether component can be used, however, optical disk baseboard in case of which birefringence (double refraction) is difficult to get generated, that has high heat resistance, satisfactory dimension stability due to low moisture

absorption property, excellent anti environment property and moreover, has sufficiently high C / N ratio and high authenticity, has not been discovered as yet.

The present invention has been devised in the view of these circumstances and offers an optical element that has low birefringence (double refraction) even by injection molding, compression molding etc., low birefringence (double refraction) with respect to the light incident from the slanting direction, high heat resistance, satisfactory balance of mechanical strength, excellent dimension stability and less foreign particles and high authenticity.

**(Method to solve the problems)**

The present invention relates to the optical element made of resin that has foreign particles of more than 1  $\mu\text{m}$  of less than 10,000 units/g.

Particularly, the present invention relates to the method for manufacture of optical element that is formed

from the resin composite formed from polymer component containing aromatic vinyl unimer units, as its main constituent and polyphenylene ether component, and that has foreign particles of more than 1  $\mu\text{m}$ , of less than 10,000 units / g.

As regards the optical element occurring in the present invention, besides optical disk baseboard, opto-magnetic disk baseboard, optic card, lens, prism etc. can be given.

In the optical disk baseboard, if foreign particles are present in large number, then C / N ratio lowers and pit errors increase, however, the fine particles that exert bad influence, are the fine particles having size of more than 1  $\mu\text{m}$  and it was clear that fine particles smaller than that do not cause any problem.

Number of units of particles of more than 1  $\mu\text{m}$  should be less than 10,000 units/g, desirably, less than 5,000 units from the point of optical disk baseboard.

If fine particles are present in a quantity of more than



that, then C / N ratio lowers and pit error increases and authenticity of optical disk becomes bad and properties lower.

The resin composite of the present invention is the mixture of polymer containing aromatic vinyl unimer units as its main constituent and polyphenylene ether, or block copolymer or graft copolymer formed from both the polymer components or their mixture.

As regards the polymer containing aromatic vinyl unimer units used in the present invention, aromatic vinyl unimer independent polymer or the copolymer containing more than 50 weight % of aromatic vinyl unimer, can be used. As regards the aromatic vinyl unimer, styrene,  $\alpha$  - methyl styrene, m - methyl styrene, p - methyl styrene, o - chloro styrene, m - chloro styrene, p - chloro styrene, m - bromo styrene, p - bromo styrene etc. can be given, however, styrene is particularly desired to be used.

Moreover, as regards the examples of unimer that is

copolymerized with aromatic vinyl unimer, unsaturated nitrile ring such as acrylonitrile, methacrylonitrile; methacrylic acid alkyl esters such as methyl methacrylate, n - propyl methacrylate, iso - propyl methacrylate, n - butyl methacrylate, cyclo hexyl methacrylate; acrylic acid esters such as methyl acrylate, ethyl acrylate, propyl acrylate, butyl acrylate etc. can be given and further, methacrylic acid, acrylic acid, anhydrous maleic acid, anhydrous citraconic acid, N - methyl maleimide, N - phenyl maleimide etc. can be given.

These unimers that are copolymerized, can be used independently or as a mixture. However, these are mixed or used within the range such that the transparency of copolymer formed with aromatic vinyl unimer, as well as resin material formed from it and polyphenylene ether, is not lost.

The aromatic vinyl unimer is desired to be contained in a quantity of more than 50 weight % in the unimer mixture.

If it is contained in a quantity of less than 50 weight %, then moisture absorbance of the obtained resin increases which is not desired.

Moreover, as regards the fusion flowability of the polymer containing aromatic vinyl unimer as its main constituent, its melt flow rate (MFR) at 230°C and load of 3.8 kg should be within the range from 0.5 ~ 200, desirably, it should be 2 ~ 100.

If it exceeds 200, then mechanical strength lowers whereas if it is less than 0.5, then it becomes difficult to lower birefringence (double refraction) which is not desired.

As regards the manufacturing method of the polymer containing aromatic vinyl unimer as its main constituent, lump state polymerization, suspension polymerization, emulsification polymerization, solution polymerization, using radical initiator, can be given, however, lump state polymerization or suspension polymerization are desired

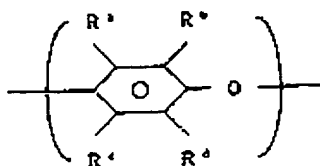
from the point of obtaining polymer having satisfactory productivity and less mixing of impurities.

As regards the radical initiator, peroxides such as lauroyl peroxide, benzoyl peroxide, di - tert - butyl peroxide, di cumyl peroxide etc., azo compounds such as 2, 2' - azo bis iso butylonitrile, 1, 1' - azo bis (1 - cyclo hexane carbonitrile) etc. can be given.

Moreover, in order to regulate the molecular weight, tert - butyl, n - butyl, n - octyl, n - do decyl and tert - do decyl mercaptan etc. can be added as chain transfer agent, as per requirement.

Polymerization is generally carried out at the temperature of 50 ~ 150°C.

Polyphenylene ether used in the present invention is the polymer possessing repetitive units shown by general formula given below.



(Here,  $R^a$ ,  $R^b$ ,  $R^c$  and  $R^d$  show hydrogen, halogen or hydrocarbon radical.)

The polyphenylene ether mentioned above is the polymer obtained by polymerizing phenol group unimer by oxidative coupling, and it is manufactured by well-known methods using copper group or manganese group catalyst, that are described in Patent number Sho 36 - 18692, Sho 47 - 36518, Sho 48 - 17396, Sho 49 - 16120, Sho 57 - 44623, Sho 57 - 147517, Sho 58 - 19329, Sho 58 - 19329, Sho 58 - 19330, Sho 58 - 122919.

As regards the concrete examples of this polyphenylene ether, poly (2, 6 - di methyl - 1, 4 - phenylene) ether, poly (2 - methyl - 6 - ethyl - 1, 4 - phenylene) ether, poly (2 - methyl - 6 - propyl - 1, 4 - phenylene) ether, poly (2,6 - di propyl - 1, 4 - phenylene) ether, poly (2 - methyl - 6 - bromo - 1, 4 - phenylene) ether etc. can be given, however, poly (2, 6 - di methyl - 1, 4 - phenylene) ether is particularly desired to be used.

Moreover, as regards this polyphenylene ether, material generally used as engineering plastic can be used, however, material having low molecular weight is appropriate.

In other words, average molecular weight of the polyphenylene ether is expressed by limiting viscosity of the polymer (measured at 25°C in chloroform) and it is desired to be within the range from 0.1 ~ 1.0 and more desirably, it should be within the range from 0.3 ~ 0.7. Further desirably, it should be within the range from 0.3 ~ 0.45 and particularly desirably, it should be within the range from 0.35 ~ 0.42.

If it is less than 0.3, then mechanical strength of the optical element lowers.

As regards the proportion of polymer containing aromatic vinyl unimer units as its main constituent and polyphenylene ether, proportion of the former should be within the range from 30 ~ 70 weight %, desirably, within the range from 40 ~ 55 weight % and the proportion of latter

should be within the range from 30 ~ 70 weight %, desirably, within the range from 45 ~ 60 weight %.

If polyphenylene ether component is less than 40 weight % or more than 70 weight %, then birefringence (double refraction) of the optical element does not lower sufficiently.

Moreover, if it less than 40 weight %, then sufficient heat resistance is not obtained.

In the resin composite, molding method is appropriately chosen within the range mentioned above.

For example, in injection molding, the proportion mentioned above is regulated so as to match the properties required by the application such as birefringence (double refraction) of the obtained molded product, corresponding to the molding conditions at the time of carrying out molding process, such as resin temperature, molding pressure, metallic mold temperature etc.

Moreover, optical element of the present invention,

particularly, optical disk baseboard permeates light of semiconductor laser beam.

Therefore, light permeability of the material having thickness of 1.2 mm at the wavelength of 200 nm, is desired to be more than 75 %.

In order to mix the polymer containing aromatic vinyl unimer units as its main constituent and polyphenylene ether to obtain resin composite of the present invention, fusion mixing or solution mixing is carried out.

As regards the extent of mixing, both the polymers are desired to be dispersed up to less than approximately 1  $\mu$  mutually and further, these are desired to be mixed up to the molecular scale.

Whether the mixing conditions are reached up to the molecular scale or not is easily determined when the mixture becomes the material having only one glass transition temperature.

Fusion mixing is carried out at the temperature above



the glass transition temperature of polyphenylene ether under high shear using mixing devices such as extruder, bombarding mixer, kneader blender, heating roll etc.

In order to obtain sufficient and satisfactory mixing conditions, the method in which mixing temperature is increased and mixing period is stretched and further, shear strength is increased, is used.

Furthermore, in the fusion mixing, organic solvent can be used in small quantity as plasticizer in order to lower the glass transition temperature of both the polymers and make the mixing easy.

As regards the organic solvent, organic solvents used in the solution mixing mentioned below can be used and after the completion of mixing, the used organic solvent can be removed by evaporation.

In order to prevent mixing of foreign particles having size more than 1  $\mu\text{m}$  from the resin composite present in the fused state, in fusion mixing, fused material

mentioned above is passed through a filter, particularly sintered metal filter.

It is passed through this sintered metal filter at high viscosity of fused resin, under high pressure and small pores.

The sintered metal filter can be tube shaped or disk shaped.

As regards the particle size, material of  $1\text{ }\mu\text{m} \sim 10\text{ }\mu\text{m}$  measured as per JIS B 8956 is used.

In order to materialize it in large industrial quantity, fusion mixing is appropriate to be carried out by screw extruder installed with sintered metal filter at its end.

The fused resin can be taken out in the form of sending it to the process of molding into optical element carried out after that.

For example, it is pelletized to be used in injection molding.

In the solution mixing, either solution of at least 1

weight % is formed by dissolving both the polymers in an organic solvent and homogeneous mixture is formed by carrying out stirring mixing and after that, organic solvent is removed by evaporation, or poor solvent of both the polymers is put in the homogeneous mixture and both the mixed polymers are deposited.

As regards the appropriate organic solvent, chloroform, methylene chloride, ethylene chloride, toluene, benzene, chloro benzene etc. can be given. Moreover, as regards the poor solvent, methanol, ethanol, propyl alcohol, n - hexane, n - pentane etc. can be given.

As regards the method of recovery of both the solution mixed polymers, besides the method of adding poor solvent mentioned above and carrying out deposition and the method of removing the solvent by evaporation, the method in which resin solution directly or resin solution formed by concentrating a part of it by ---, is taken in a screw extrusion crusher and pelletization is carried out

simultaneously while removing the solvent, or the method in which organic solvent is azeotropically removed by adding water to the resin solution and aqueous slurry of the resin powder is formed and recovery is carried out, can be used.

In order to remove the foreign particles of more than 1  $\mu\text{m}$  from the solution mentioned above by solution mixing, the solution mentioned above is filtered beforehand.

The filter material used at the time of filtration is required to remove foreign particles of more than 1  $\mu\text{m}$  and as regards such type of filter material, filter paper, bulb, sintered metal, sintered material of metal ---, ceramic filter material etc. can be given.

Filter paper and sintered material of metal ---- are desired to be used as filter material.

As regards the filtration device, batch type such as pressure filter, elevated pressure filter, vacuum filter etc., or common type of filters of continuous type can be

used.

Temperature at the time of filtration is desired to be 60°C ~ 130°C in order to lower the viscosity of the solution mentioned above.

The solution mentioned above obtained after filtration is treated as mentioned below and the resin composite is recovered.

Fusion molding in the present invention means the molding carried out at the resin temperature above the glass transition temperature, in the state of ---.

For example, extrusion molding, compression molding etc. can be used.

Molding temperature is of the order of 350°C above the glass transition temperature of the resin.

Injection molding, compression molding, extrusion molding etc. can be used as the molding method at the time of obtaining optical element of the present invention, however, among these molding methods, injection molding

is the most desired from the point of degree of birefringence (double refraction) generated by molding and productivity.

The injection molding stated here is the method to manufacture molded product that is cooled to solidification by pressing the resin present in fluid state by heating the opened metallic mold cavity.

Moreover, method of vacuum suction in metallic mold or the injection compression method in which capacity of metallic mold cavities is reduced is used in the injection molding.

At the time of manufacturing optical element of the present invention by injection molding, injection molding is desired to be carried out at the fusion plastized resin temperature of more than  $270^{\circ}\text{C}$  and less than  $350^{\circ}\text{C}$ , desirably, more than  $300^{\circ}\text{C}$  and less than  $340^{\circ}\text{C}$ .

Resin temperature stated here is the temperature of resin present inside the injection cylinder that is

plastized and fused by the heat generated by external heating of motor and shear that is generated by rotation of screw, in the injection molding machine.

If the resin temperature is less than  $270^{\circ}\text{C}$ , then birefringence (double refraction) of the optical element, particularly optical disk baseboard becomes more than 20 nm and it cannot be used as optical disk baseboard. On the other hand, if the resin temperature exceeds  $350^{\circ}\text{C}$ , then resin gets decomposed and undesired phenomenon such as burning, silver etc. get generated and pit errors of the obtained optical disk baseboard conspicuously increase which is not desired.

In this injection molding, metallic mold temperature is desired to be more than  $50^{\circ}\text{C}$  and less than  $140^{\circ}\text{C}$  and more desirably, it should be more than  $80^{\circ}\text{C}$  and less than  $120^{\circ}\text{C}$ .

Metallic mold temperature stated here means surface temperature of metallic mold cavity just before carrying out injection.

If the metallic mold temperature is less than 50°C, then transcription property of the fine groove engraved on the surface of metallic mold becomes bad whereas if it exceeds 140°C, then release of the molded material from the metallic mold becomes bad.

In the injection molding of the present invention, injection molding period is desired to be more than 0.2 seconds and less than 3 seconds, more desirably, it should be more than 0.3 seconds and less than 2 seconds.

The injection molding period stated here means the period of filling the resin in the metallic mold cavity.

If the injection molding period is less than 0.2 seconds, then silver gets generated and in the case of using it as optical disk, pit errors conspicuously increase whereas if it exceeds 3 seconds, then birefringence (double refraction) of the optical disk baseboard becomes more than 20 nm which is not desired.

Particularly, the method of making the foreign



particles of more than 1  $\mu\text{m}$  present in the resin composite formed from the polymer component containing aromatic vinyl unimer units as its main constituent and polyphenylene ether component, less than 10,00 units / g, is as described above, however, different devices are further required in order to prevent the mixing of foreign fine particles.

For example, as regards the raw material used for the manufacture of resin (monomer, polymerization initiator, solvent etc.), material containing no foreign fine particles are used.

Manufacture device is cleaned as long as possible and moreover, adhesion of foreign substance is prevented.

In order to prevent mixing of foreign substance in molding process, environment is kept clean to the extent possible.

Particularly, after removing foreign fine particles, it (molding) is desired to be carried out in clean

atmosphere.

**(Practical examples)**

The present invention has been explained below in further details with the help of practical examples. However, the present invention is not restricted only to these practical examples.

Moreover, in the practical examples, "parts" or "%" are weight standards.

Moreover, measurement of properties indicated in practical examples and procedures were carried out by following methods.

- Birefringence (double refraction): Retardation was measured by Celmon (?) Compensator method at 546 nm.

- Water absorbance: Average water absorbance was measured in hot water of 60°C as per ASTM D-570.

- Light permeability: Permeability of test material having thickness of 1.2 mm was measured at 800nm by spectrophotometer of Hitachi make 330 model.

- Bending property: It was measured by ASTM D-790.
- Heat resistance: It is expressed by glass transition temperature measured by coefficient of linear expansion (?) method.
- Limiting viscosity of polymer: It was calculated by using Ubelhode viscometer over chloroform solution at 25°C.
- C / N ratio: It is the value measured by using spectrum analyzer 353A of Hewlett Packard company under the conditions of no difference effect (?) optics system and band width of scanning filter of 30 KHz.
- Number of foreign fine particles: Number of foreign fine particles per 1 g of test material was measured by measuring units of particles of more than 1  $\mu\text{m}$  by automatic fine particles measuring device KL - 01 model of Lion company.
- Mixing and pelletization was carried out by biaxial screw extruder (Nippon Seiko Kabushiki Kaisha (Japan

Steel Co. Ltd.) make, TEX 30 - 30 BW - 2 V).

· Injection molding was carried out by using Sumitomo Jukikai Kogyo Kabushiki Kaisha (Sumitomo Heavy Machinery Industries Ltd.) make Neomat 150 / 75 (75 ton) as injection molding machine and metallic mold used in disk having diameter of 120 mm and thickness of 1.2 mm as metallic mold.

· Manufacturing method of optical disk baseboard: Reactive sputtering of silicon is carried out on the baseboard obtained by injection molding under the reduced pressure of  $5 \times 10^{-3}$  Torr in the atmosphere of mixed gas of argon and nitrogen and silicon nitride thin film having refractive index of 2.0 and thickness of 850 Å was obtained.

· Furthermore, magnetic film of TbFeCo was attached on it with the thickness of 900 Å by sputtering method and moreover, silicon nitride film mentioned above was attached with the thickness of 850 Å and thus, optical disk

possessing structure of baseboard / silicon nitride film  
/ TbFeCo / silicon nitride film was prepared.

#### **Practical example 1**

As per the method described in practical example 2, No.  
9 of Patent number Sho 47 - 36518, poly (2, 6 - di methyl  
- 1, 4 - phenylene) ether having limiting viscosity of 0.52  
was prepared by polymerizing 2, 6 - xylenol using  
manganese chloride and ethanol amine as solvents.

40 parts by weight of this polyphenylene ether and 60  
parts by weight of polystyrene resin (Sumitomo Kagaku  
Kogyo (Sumitomo Chemical Industries) make) Esbright (?)  
2, normal grade, MFR 10) were mixed and blended and were  
kneaded and pelletized by using a biaxial screw extruder  
installed with gear pump and disk type sintered metal  
filter of Nippon --- (Japan --- company) having filtration  
particle size of 3  $\mu$ m standardized by JIS B 8356, at the  
end part.

The pellet mentioned above was extrusion molded at the

cylinder temperature of 320°C and metallic mold temperature of 85°C and an optical disk baseboard having diameter of 120 mm and thickness of 1.2 mm was prepared.

Light permeability of this disk baseboard was 86 %, birefringence (double refraction) at the position of 35 mm from the center of the round plate was + 2 nm, water absorbance was 0.1 %, glass transition temperature 143°C and number of foreign fine particles was 7,000 units.

C / N ratio of the optical disk was 45 dB.

#### **Comparative example 1**

At the time of fusion pelletization, disk type sintered metal filter was not installed. Other than this change, procedure same as that of practical example 1 was carried out and optical disk baseboard was obtained.

Number of foreign fine particles present in the obtained optical disk baseboard was 12,000 units / g and C / N ratio of the optical disk was 39 dB.

Moreover, foreign substance was visually observed under

microscope.

## **Practical example 2**

50 parts by weight of polyphenylene ether having limiting viscosity of 0.40 and 50 parts by weight of polystyrene resin (Sumitomo Kagaku Kogyo (Sumitomo Chemical Industries) make) Esbright (?) 4 - 62A) were dissolved in 400 parts by weight of chloroform after which stainless --- filter (Nippon --- (Japan ---- company) make, Naslon (?) NP103) was used in --- material and filtration was carried out under elevated pressure of 20 kg / cm<sup>2</sup>G.

The resin solution mentioned above was dropped in 2000 parts by weight of methanol and resin was precipitated and the obtained resin was pelletized.

The pellet mentioned above was injection molded at the resin temperature of 320°C, metallic mold temperature of 100°C and injection period of 1 second, and an optical disk baseboard was prepared.

Birefringence (double refraction) of this baseboard, at

the position of 40 nm from the center was - 2 nm and number of foreign fine particles were 1,000 units / g.

Moreover, bending strength of the resin was 1100 kg / cm<sup>2</sup> and glass transition temperature was 154°C.

C / N ratio of the optical disk was 47 dB.

### Practical example 3

44 parts by weight of polyphenylene ether having limiting viscosity of 0.44 and 56 parts by weight of polystyrene (Nippon Polystyrene Kogyo Kabushiki Kaisha (Japan Polystyrene Industries Ltd.) make, Esbright (?) 4 - 62A) were dissolved in 300 parts by weight of toluene that was heated at 80°C and stainless --- filter (Nippon --- (Japan ---- company) make, Naslon (?) NP103) was used in filter material and filtration was carried out under elevated pressure of 10 kg / cm<sup>2</sup>G.

The resin solution mentioned above was dropped in 1000 parts by weight of water and it was stirred. After this, toluene was removed and aqueous slurry of resin was



obtained.

This slurry was centrifugally separated and resin was obtained.

Other than this change, procedure same as that of practical example 2 was carried out and evaluation was carried out.

Birefringence (double refraction) of the baseboard was + 1 nm and number of foreign fine particles were 2,000 units / g.

Moreover, bending strength of the resin was 1200 kg / cm<sup>2</sup> and glass transition temperature was 148°C.

C / N ratio of the optical disk was 46 dB.

**(Effect / result of the invention)**

If the present invention is used, then optical disk baseboard having high authenticity can be obtained due to having foreign particles of more than 1  $\mu$ m of less than 10,000 units/g.

Particularly, the optical element formed from polymer

component containing aromatic vinyl unimer units, as its main constituent and polyphenylene ether component, has foreign particles of more than 1  $\mu\text{m}$  present in the resin composite mentioned above are less than 10,000 units / g due to which optical element having high properties and high authenticity such as excellent optical isotropy, high heat resistance can be obtained.

Optical element of the present invention is optical disk baseboard, optical card, lens, prism and it can be appropriately used in the case of using light possessing specific wavelength.

Furthermore, as mentioned above, an optical disk having low birefringence (double refraction), high heat resistance, satisfactory mechanical properties and moreover, having sufficiently high C / N ratio at the time of recording and regeneration, can be obtained.